Effect of carrier type on the spray-dried willowherb (*Epilobium angustifolium* L.) leaves extract, powder properties and bioactive compounds encapsulation

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Willowherb (*Epilobium angustifolium* L.) leaves are a valuable source of polyphenolic compounds (phenolic acids, flavonoids, and ellagitannins) that are mainly used in the treatment of benign prostatic hyperplasia. In the presented study, the impacts of maltodextrin and whey protein as carriers on the efficiency of spray drying and physicochemical properties of the obtained powders were examined. The use of carriers significantly improved the drying yield (over 60 %). Moisture content, hygroscopicity, and rehydration time of all dried extracts (without the carrier, with maltodextrin and whey protein) were at an acceptable level, while their flowability and cohesiveness were poor. The encapsulation efficiency of polyphenols in a whey protein carrier of 92.02 % was significantly higher compared to 75.80 % for maltodextrin. These results show that whey protein efficiently encloses the extract ingredients into powder particles and thus preserves sensitive phenolic compounds during the drying process. The efficacy of flavonoid encapsulation was also high (93.00 % for maltodextrin and 94.34 % for whey protein), with no statistically significant differences between carrier types. Therefore, willowherb leaves extract can be successfully encapsulated by spray drying using maltodextrin and whey protein as carriers.

Key words: Epilobium angustifolium; willowherb; microencapsulation; wall materials; spray dry; phenolics

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1. INTRODUCTION

The genus *Epilobium* (Onagraceae) encompasses approximately 200 plant species distributed worldwide. Among them, one of the best known medicinal plants is *Epilobium angustifolium* L. (syn. *Chamaenerion angustifolium* (L.) Scop.), commonly named willowherb or fireweed (Granica et al., 2014). In folk medicine, teas of willowherb are mostly used in the treatment of urinary tract diseases, such as micturition, benign prostatic hyperplasia, prostatitis, and urethral inflammation. Apart from urinary disease, the willowherb is traditionally used to treat wounds and skin diseases, pain (migraine headaches, childbirth, stomach ache, sore throat), menstrual disorders, gastrointestinal disorders, insomnia, fever (Granica et al., 2014; Schepetkin et al., 2016; Shikov et al., 2006; Sõukand et al., 2020). The justification for its use to reduce nocturia in subjects with benign prostatic hyperplasia was confirmed by clinical trial (Esposito et al., 2021). The main pharmacologically active ingredients of willowherb extracts are polyphenols including phenolic acids, flavonoids and ellagitannins. The dominant secondary metabolite in all *Epilobium* species and main active principle is the macrocyclic ellagitannin oenothein B (Hevesi Tóth et al., 2009). The dominant flavonoid in *E. angustifolium* is miquelianin (quercetin-3-O-glucuronide) unlike other *Epilobium* species where the predominant glycosides are derivatives of kaempferol and myricetin. *E. angustifolium* is a rich source of phenolic acids such as ellagic acid, chlorogenic acid, benzoic acid, trans-cinnamic acid and their derivatives (Granica et al., 2014; Schepetkin et al., 2016; Stolarczyk et al., 2013).

Instability in an environment of high humidity, light and oxygen, unpleasant astringent taste and low bioavailability of polyphenolic compounds can significantly affect the therapeutic efficacy of extracts. These limitations could be solved using encapsulation techniques such as spray drying (Ćujić Nikolić et al., 2018). By encapsulation, the liquid form of the extract is converted into a powder in which the active ingredients are trapped in the coating of the wall materials (carriers). Encapsulation improves the processing and storage stability as well as release control and targeted drug delivery, enables

easier handling and masking unpleasant tastes. The obtained powder can be used as a final product (instant tea) or as a semi-finished product for further processing and incorporation into advanced drug formulations (Bakowska-Barczak and Kolodziejczyk, 2011; Ballesteros et al., 2017; Mahdi et al., 2020). Spray drying as a quickly, uncostly, affordable and flexible technique is suitable for large-scale powders production in the pharmaceutical and food industry with acceptable encapsulation efficiency (Chaumun et al., 2020). Choosing the optimal carriers is one of the key steps in the development of the appropriate encapsulation process. The carriers should provide maximum incorporation and retention of the extract ingredients, to create a protection against damaging impact, to be biodegradable, safe and compatible with active compounds. Carbohydrate- and protein-based natural biopolymers, including maltodextrin (MD) and whey protein (WP) are the most commonly used carriers for encapsulation via spray drying (Mahdi et al., 2020). To our knowledge, no encapsulation study of *E. angustifolium* extract by spray drying has been performed. The aim of presented study was to evaluate the encapsulation efficiency of polyphenolic compounds from *E. angustifolium* leaves using spray drying technique with two different, commonly used, carriers (maltodextrin as carbohydrate carrier and whey protein as protein carrier) and to characterize the encapsulated powders.

2. MATERIALS AND METHODS

2.1. Chemicals

Maltodextrin (DE16-19.9) was provided from Davisco Foods International (Le Sueur, MN, USA) and whey protein was provided from Polmlek (Raciąż, Poland). Folin-Ciocalteu reagent, gallic acid, catechin, and sodium carbonate were purchased from Sigma–Aldrich Chemie GmbH (Munich, Germany). All other chemicals used in this study were of analytical reagent grade.

2.2. Plant material and preparation of liquid extract

Willowherb (*Epilobium angustifolium* L.) leaves were obtained from the Institute for Medicinal Plants Research "Dr. Josif Pančić". Liquid extract was prepared by decoction with solid to solvent ration 1:20. After extraction, extract was immediately filtered through the filter paper with pore size 4-12 μm (Schleicher & Schuell, Dassel, Germany). Obtained liquid extract was collected into the glass flask and used for future experiments.

2.3. Spray drying process

Liquid willowherb extract (LE) was spray dried with and without carrier addition. Two different biopolymers, MD and WP, in concentrations of 20 % were used. Each biopolymer was separately dissolved in a previously produced LE, and the concentration used in experiments were calculated based on the dry weight of the LE. The prepared solutions were heated at 40 °C and mixed using magnetic stirrer to completely homogenization, before the spray drying process. The liquid feed was spray dried in a Labtex ESDTi spray dryer (Labtex, Huddersfield, UK) with 0.5 mm standard diameter nozzle under following conditions: inlet temperature 130±5 °C, outlet temperature 80 ± 5 °C, spraying air flow rate (75 m^3/h), liquid feed (10.8 mL/min rate), atomization pressure (2.6 bar). The obtained spray-dried willowherb extract was separated from the air by a cyclone. Free-flowing powders were obtained and transferred to high-density glass bottles before analyses. They were stored in the dark, in desiccator at room temperature, and these conditions ensured physical stability and active compounds preservation.

2.4. Powder yield

The yield (Y) of drying process was calculated as the ratio between mass (g) of the spray-dried extract and the expected mass:

$$Y\,[\%] = \frac{m_{extract}}{m_{expected}} \times 100 \quad (1)$$

Expected mass was calculated as the sum of share of dry residue in LE multiplied with a mass of LE used for drying process and mass of the used carrier:

$$m_{expected}\,[g] = m_{dry\,residue} \times m_{LE} + m_{carrier} \quad (2)$$

2.5. Physical characterization of powders

2.5.1. Moisture content

The moisture content of each sample was analyzed thermo gravimetrically. The collected powders were dried at 105 °C until they achieved constant weight using Halogen Moisture Analyzer HB43-S by Mettler Toledo. The results are expressed as a percentage (%) relative to the dried extract.

2.5.2. Flowability properties

For the determination of bulk density, 2 g of powder was gently loaded into a 10 ml graduated cylinder. The measured volume read directly from the cylinder was used to calculate the bulk density (ρ_{bulk}) according to the ratio of mass to volume. In order to determine the tapped density (ρ_{tapped}) of extract powders, the cylinder was tapped for 120 times and the volume of the sample was read (Jinapong et al., 2008). Flowability and cohesiveness values of the powders were evaluated in terms of Carr index (Carr, 1965), and Hausner ratio (Hausner, 1967), respectively. Both, CI and HR, were calculated from the bulk (ρ_{bulk}), and tapped (ρ_{tapped}) densities of the powder by using next equations:

$$Carr\ index = \frac{\rho_{tapped} - \rho_{bulk}}{\rho_{tapped}} \times 100 \quad (3)$$

$$Hausner\ ratio = \frac{\rho_{tapped}}{\rho_{bulk}} \quad (4)$$

2.5.3. Hygroscopicity

Hygroscopicity of powders was determined according to the modified method of (Cai and Corke, 2000). Approximately 1 g of obtained powder was placed at room temperature in stability chamber (Memmert, Schwabach, Germany), filled with NaCl saturated solution (70 % RH). Hygroscopicity was monitored during 7 days. Results were expressed in percent (%), and calculated as gram of absorbed water (moisture) per 100 g of powders (g/100 g).

2.5.4. Rehydration

Rehydration time of powders is a period during the dry extract is completely dissolved in water at room temperature. Tests were carried out on magnetic stirrer, and it has been measured the time taken to fully reconstitute 1 g of powder in 50 mL of water, expressed in seconds (s) (Goula and Adamopoulos, 2010).

2.6. Chemical characterization of powders

2.6.1. Total phenolic content

For TP determination, Folin-Ciocalteu assay with slight modifications was applied (Waterman and Mole, 1994). The reaction mixture was prepared by mixing 200 μL of each sample and 1000 μL of 10 % Folin-Ciocalteu reagent and after four minutes 800 μL of 7.5 % Na_2CO_3 was added. The mixture was incubated for 2 hours. Distilled water was used as blank, while control was prepared to contain distilled water instead

Table 1. Production yield and physical characterization of *E. angustifolium* powders.

Extracts	Powder yield[a] [%]	Moisture content [%]	Rehydration [s]	Bulk density [mg/mL]	Tapped density [mg/mL]	Carr index	Hausner ratio
Without carrier	47.75	4.11±0.63 a	20.00±1.21 a	131.60±0.40 c	227.30±0.22 b	42.11±0.23 b	1.73±0.01 b
With 20 % MD[b]	63.76	2.43±0.29 ab	30.00±2.00 b	116.30±0.21 a	185.20±0.13 a	37.21±0.14 a	1.59±0.00 a
With 20 % WP[c]	63.18	2.04±0.04 b	30.00±2.87 b	125.00±0.11 b	227.30±0.15 b	45.00±0.20 c	1.82±0.01 c

[a] Different letters (a-c) in each column showed a significant difference (P<0.05) between different extracts according to the *post-hoc* Tukey test.
[b] MD - stands for maltodextrin
[c] WP - stands for whey protein

of sample. Absorbance was recorded at 740 nm after two hours incubation at room temperature. Obtained results were presented as milligrams of gallic acid equivalent per gram of powders (mg GAE/g).

2.6.2. Total flavonoid content

The content of total flavonoids (TF) was determined by the aluminum chloride spectrophotometric method described by (Loizzo et al., 2012). The sample absorption was measured at a wavelength of 510 nm. Based on the calibration curve of a standard catechin solution, the content of total flavonoids was determined. The total flavonoid content was expressed as mg of catechin equivalent per g dry extract (mg CE/g). All experiments were replicated three times and results were expressed as mean values.

2.6.3. Encapsulation efficiency

The encapsulation efficiency (EE %) for microencapsulated powders were calculated according to the equation:

$$EE\,[\%] = \frac{E}{E_{total}} \times 100 \quad \textbf{(5)}$$

where E represents quantity of TP or TF microencapsulated in the powders, and E_{total} presents quantity of TP or TF and their respective amount in the LE.

2.7. Statistical analysis

All experiments were executed in triplicates determinations. Results were presented as mean value ± standard deviation. One-way ANOVA was conducted to test the individual factors influence on observed property and Tukey post hoc test was used for differences between the mean values detection. Significant levels were considered at P<0.05 (STATISTICA v.7.0.3). Statistical analysis was performed using the MS Office Excel v. 2010.

3. RESULTS

3.1. Production yield

Powder yield was defined as the ratio of the experimentally obtained powder and the gravimetrically calculated dry residue of the liquid extract. From an industrial point of view, maximizing production yield is one of the essential criteria when choosing a drying aid, as it is closely related to production costs and efficiency. A spray drying process is considered to be of acceptable efficiency if the production yield is greater than 50 % (Vidović et al., 2014). In our study, significant adhesion of particles to the walls of the drying chamber was observed during the drying process of the extract without carrier. Measured yield of 47.75 % for *E. angustifolium* dry extract without carrier was slightly lower than the mentioned limit, while the yields of powders with MD and WP (63.76 and 63.18 %, respectively) were significantly higher. Obviously, an improvement in the production yield was achieved by the addition of carriers. The reduction of the stickiness of the particles for the chamber walls and the consequent increase of the yield by adding the carriers is in accordance with the results of (Vidović et al., 2014).

The main mechanism of powder yield improvement using carbohydrate carriers such as MD is to increase the glass transition temperature of the feed mixture, thus reducing the particle stickiness. On the other hand, WP does not affect the glass transition temperature but affects the properties of film formation. Actually, WP as a surfactant can migrate to the droplet surface and accelerate the formation of a glassy film in a heated air stream that prevents particles from interacting with the chamber wall (Tontul and Topuz, 2017).

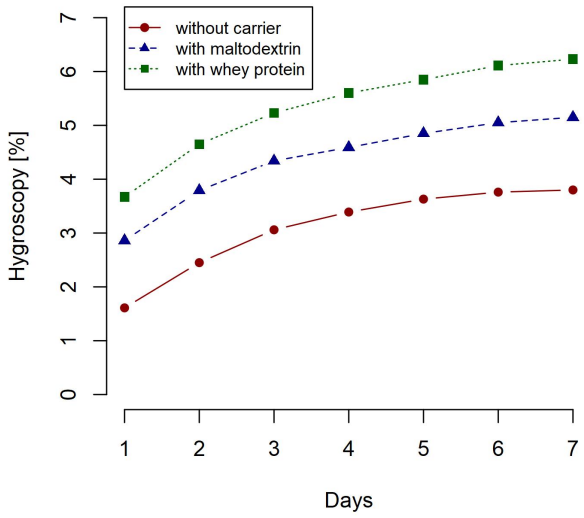

Fig. 1. Hygroscopicity of spray-dried *E. angustifolium* leaves extract.

3.2. Physical properties of *E. angustifolium* powders

The mean values of the examined physical properties for the obtained powders are shown in Table 1. The residual moisture content of 2.43 and 2.04 % in powders with MD and WP, respectively, was significantly lower compared to 4.11 % in powder without carrier. This is in line with the literature data showing that the moisture content of the spray-dried powder decreases with increasing proportion of the carrier materials in the feed mixture. Low residual moisture in the powder is closely related to the improvement of microbiological stability

Table 2. Content and encapsulation efficiency of total phenolics and total flavonoids in *E. angustifolium* extracts.

	Total phenolics		Total flavonoids	
Extracts	Content[a] [mg GAE/g]	Encapsulation efficiency [%]	Content [mg CE/g]	Encapsulation efficiency [%]
Liquid extract	448.7±9.78 c	-	131.2±0.85 b	-
Extracts without carrier	342.6±1.65 a	-	130.7±0.50 b	-
Extracts with 20% MD[b]	340.1±1.79 a	75.80±0.40 a	122.0±1.10 a	93.00±0.82 a
Extracts with 20% WP[c]	412.9±0.75 b	92.02±0.17 b	123.8±1.25 a	94.34±0.96 a

[a] Different letters (a-c) in each column showed a significant difference (P<0.05) between different extracts according to the *post-hoc* Tukey test.
[b] MD - stands for maltodextrin
[c] WP - stands for whey protein

and the consequent prolongation of the extract shelf-life. If the moisture content is less than 5 %, the powder can be classified as microbiologically stable. Apart of microbiological stability, the moisture affects the rheological properties of the powder. Namely, moisture acts as a plasticizer due to the depression of the glass transition temperature and affects the caking properties during storage (Tontul and Topuz, 2017; Vidović et al., 2014).

The hygroscopicity of the powders was measured for a week under conditions of high relative humidity. As expected, the largest increase in absorbed moisture in all examined powders was recorded after the first 24 hours with a slight gradual increase in the following period (Figure 1). On the seventh day, hygroscopicity of 3.8, 5.15 and 6.23 % was recorded for powders without carriers, with MD and WP, respectively. Since the observed hygroscopicity of the powders were significantly lower than 20 %, they can be considered as poor hygroscopic (Tontul and Topuz, 2017). Interestingly, an inverse relationship between the powder moisture content and its hygroscopicity was observed. The highest hygroscopicity of WP powder can be explained by the high water-holding capacity of the amorphous protein carriers (Tontul and Topuz, 2017).

Instant powder rehydration without lumps and sediments formation is an important benchmark of quality for the end-users. The main strategy for improving the rehydration powder properties is using the spray drying method. The relatively short rehydration time of 20 s for powder without carrier and 30 s for powders with MD and WP is satisfactory. Slightly longer rehydration times of extracts with carrier may be associated with additional time required to disassemble the formed cross-links between biopolymer molecules (Hogekamp and Schubert, 2003).

Assessment the bulk and tapped (compacted) density is relevant for powders used to formulate limiting volume dosage forms, such as tablets or capsules. The highest value of bulk density was recorded in powder without carrier (131.6 mg/mL), followed by powder with WP (125.0 mg/mL) and MD (116.3 mg/mL). A similar trend, the highest bulk density for carrier-free Hibiscus sabdariffa extract and slightly lower for extracts with WP and MD was reported by (Díaz-Bandera et al., 2015). The flowability and cohesiveness of the obtained powders were estimated using Carr index and Hausner ratio, which were calculated via bulk and tapped densities. Carr index values of all powders belonging to the range from 35 to 45 indicate bad flowability, possibly due to its stickiness. Also, Hausner ratio values greater than 1.4 for all powders showed their high cohesiveness. Both the Carr index and the Hausner ratio decreased with the addition of MD, indicating that the addition of maltodextrin improved flow properties. The effect of higher concentrations of maltodextrin on flowability properties should be investigated in future studies. The highest Carr index and Hausner ratio recorded in the case of the dried extract with WP can be attributed to the highest hygroscopicity of this powder. Namely, the binding of ambient water increases the cohesiveness between the powder particles and consequently decreases the powder flowability (Caliskan and Dirim, 2016).

3.3. Encapsulation efficiency

Encapsulation efficacy was defined as the ratio of the total phenolic/flavonoid content of the experimentally obtained dry extracts and their total content in the liquid extract, expressed in percentage. Results of total phenolic content, total flavonoid content and their encapsulation efficiency in *E. angustifolium* extracts are presented in Table 2. The content of total phenolic was highest in the dry extract with 20 % WP as a drying agent (412.9 mg GAE/g). The content of total polyphenolics in the dry extract with the addition of 20 % MD (340.1 mg GAE/g) was slightly lower compared to the dry extract without carriers (342.6 mg GAE/g). The superior encapsulation efficiency of WP (92.02 %) compared to MD (75.80 %) can be partly explained by the possibility of complexation between phenolic compounds and WP molecules (de Morais et al., 2020). These results show that WP carrier effectively encloses the extract ingredients into powder particles and thus preserves sensitive phenolic compounds.

The contents of total flavonoids in dry extracts with the addition of 20 % MD and WP (122.0 and 123.8 mg CE/g, respectively) were slightly lower compared to the dry extract without carriers (130.7 mg CE/g), probably due to the dilution effect. There were no statistically significant differences between WP (94.34 %) and MD (93.00 %) as carriers in the encapsulation efficiency of flavonoid compounds.

CONCLUSION

Willowherb (*Epilobium angustifolium*) leaves extract was successfully encapsulated by spray drying employing maltodextrin and whey protein as carrier agents. Both carriers significantly improved the drying yield. Dry extracts are characterized by high encapsulation efficiency of polyphenols (over 75 %) and flavonoids (over 90 %), with the superiority of whey protein carrier in this respect. Moisture content below 5 %, as well as hygroscopicity that was significantly lower than 20 % for all obtained powders, promise desired extracts stability. The short rehydration time in an aqueous medium (about 20 s for carrier-free extract and 30 s for both extracts with carriers), without lumps and sediments, indicates that the obtained dry extracts are acceptable as instant teas. The poor flowability and cohesiveness of the carrier-free extract were slightly

improved only by maltodextrin. Therefore, spray drying of willowherb leaves extract with maltodextrin and whey protein as carriers can be successfully carried out, providing the preservation of phenolic ingredients.

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